



# Thermal diffusion profile by NRA of Deuterium implanted in solid breeder blanket materials

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## State of the art and objectives

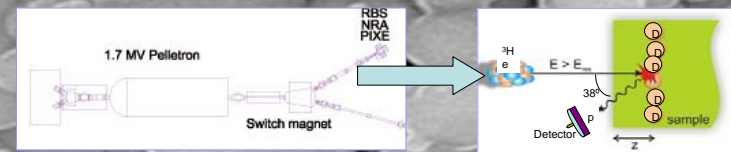
Breeder blanket materials have to produce tritium from lithium while fulfilling several strict conditions. In particular, when dealing with materials to be applied in fusion reactors, one of the key questions is the study of light ions retention, which can be produced by transmutation reactions and/or introduced by interaction with the plasma. In ceramic breeders the understanding of the hydrogen isotopes behaviour and specially the diffusion of tritium to the surface is crucial. Moreover the evolution of the microstructure during irradiation with energetic ions, neutrons and electrons is complex because of the interaction of a high number of processes.

Even when Li-ceramics are not a new issue, the old generation of ceramics presented a big handicap which so far has hampered them to be considered for fusion reactors. This was related with their high T retention. Therefore, a large effort has been done in order to develop new ceramics with a much lower T retention. Since tritium is very radioactive and difficult to handle, the most part of the experiments concerning this topic have been carried out by analysing the H and D performance, a very much similar behaviour is expected for T.

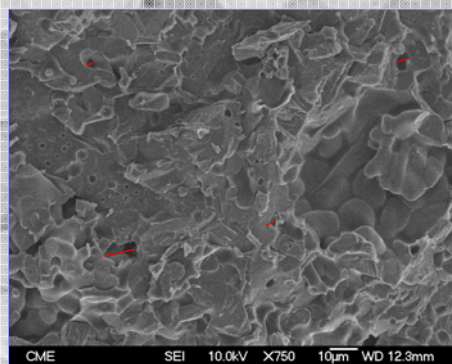
In this work we characterised the temperature dependence of D behaviour in different types of Li-based ceramics. To this aim diffusion profiles have been measured by Resonance Nuclear Reaction Analysis (RNRA) by using the  $D(^3\text{He},p)^4\text{He}$  reaction in Li-based ceramics which present different microstructure, chemical composition and Li contents.

## Experimental

- Li-based ceramics  $\text{Li}_2\text{TiO}_3$ ,  $\text{Li}_4\text{SiO}_4$ ,  $\text{Li}_6\text{SiO}_5$  were fabricated at CIEMAT:  $\text{Li}_4\text{SiO}_4$ ,  $\text{Li}_6\text{SiO}_5$  were sintetized in our laboratory by  $\text{SiO}_2$  gel + lithium citrate;  $\text{Li}_2\text{TiO}_3$  was prepared using commercial powder. Powders were then isostatically pressed at 2.5MPa, and sinterized at different Temperatures achieving residual porosity of about 20% TD.
- Samples were implanted at room temperature with D at a fluence of  $1 \times 10^{17}$  ion/cm<sup>2</sup> at the implantation facility in UCM, Spain. The implantation energy was selected to be 100keV which according to SRIM calculations corresponds to depths of ~1.6-2.4  $\mu\text{m}$ , depending on sample density.
- After implantation, samples were stored under environmental conditions for 3 days.
- Microstructure were characterised prior to and after implantation by SEM and STEM.
- The thermal dependence of D behaviour was analysed by RNRA using the  $D(^3\text{He},p)^4\text{He}$  reaction. Samples were in-situ thermal annealed at 100°C, 150°C and 200°C. RNRA experiments were carried out in Leuven KUL's laboratories, Belgium.



## Microstructural characterisation



SEM fresh fracture microstructure of a  $\text{Li}_2\text{TiO}_3$  ceramic pellet. Transgranular fracture with monophasic polygonal grains of about  $10\mu\text{m}$ . Highly porous microstructure of pore size ranging from  $1\mu\text{m}$  to  $10\mu\text{m}$



Cross sectional SEM image of an as-implanted  $\text{Li}_2\text{TiO}_3$  lamella prepared by FIB.

As expected, an amorphous layer which extends ~1  $\mu\text{m}$  underneath the sample surface is observed corresponding to the ion-implanted region whose maxim is located at aprox 300 nm.

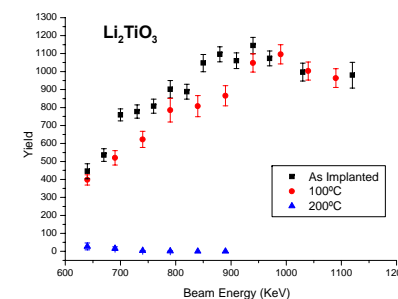
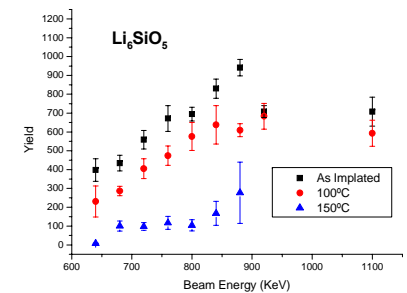
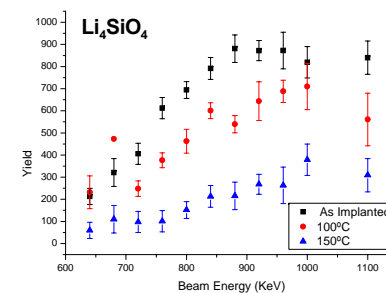
## Conclusions

- In general, temperature favors D release for all analyzed samples.
- For the sample annealed at  $T=200^\circ\text{C}$  practically all the D has been released. This result is very relevant since it might indicate that the new generation of ceramics seems to have overcome the long standing problem of H-isotopes retention.
- Further work has to be done in order to mimic the H-isotopes behavior in more realistic reactor operation conditions.

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## Thermal dependence of the D behaviour



• The yield decreases with increasing annealing temperature which indicates that D is being released from the samples.

• At  $T=200^\circ\text{C}$  the most part of the D has been already released.